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INSULATING LAYERS FOR CROSSOVERS AND MULTILAYER CIRCUITS IN HYBRID MICROSYSTEMS (PART II)

By

Danuta Lusniak-Wojcicka, Olgierda Sztaba, Leszek Golonka

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Insulating Layers for Crossovers and Multilayer Circuits in Hybrid Micro-Systems (Part II) by D. Lusniak-Wojcicka, O. Sztaba, L. Golonka

The first part of the article (PIE Papers, 1975, No. 2) discussed the role of insulating layers and presented overall requirements which these layers should fulfill as used in hybrid microsystems. The second part presents the course and results of technological tests on the usefulness of glasses having compositions rated as materials for insulating pastes. Two groups of glass with crystallization capabilities are rated for individual testing. The temperature relationship of the tangent of the angle of loss and capacity, the dielectric constant and loss in the frequency function and time function are defined. The complete usefulness of these glasses to insulate conductive paths is confirmed.

Technological tests, having for their purpose the preliminary determination of the usability of glasses having typified compositions (Part I, tables 1 and 2) as the parent substance for insulating pastes used for cross-overs and multilayer circuits, consisted in:

- the melting of glasses according to given compositions,
- disintegrating them into granules less than 40 μm,
- preparing insulating pastes using powders of the above mentioned glasses with the addition of an organic agent (8% solution of ethyl cellulose in a β terpineol),
- applying by the paint-screen method (screen with a 260 mesh gauge) the prepared insulating paste to test plates containing lower conductive paths of PdAg or Au pastes,
- heat treatment of insulating layers in a continuous furnace, during which the maximum temperature of the heat treatment was 850^{o}C, and the total time was 30 minutes,
- applying by the paint-screen method PdAg or Au pastes accordingly to upper conductive paths,
- heat treatment of upper conductive paths, during which the maximum firing temperature was 850^{o}C.

It should be noted that the fundamental parameters of the technological process such as gauge of the screen used in the paint-screen method, the maximum firing temperature, and the time of heat treatment, were compatible with the technological parameters recommended in the DuPont information.

Visual inspection of the test plates indicated first of all the usefulness of No. 4 and No. 9 glass as parent substances for insulating pastes to be used for crossovers and multilayer circuits.

The criteria of the preliminary evaluation were:

- continuity, finish and homogeneity of the surface of the insulating layer after heat treatment,
- reproducibility of the assumed configuration of the insulating layer (above all, size and shape of the openings),
- continuity and degree of reproducibility of the configurations of the upper conductive paths.

Layers from insulating pasts on the basis of the remaining glasses detailed in Tables 1 and 2 (Part I) had a series of flaws which will be briefly discussed. Thus, in the case of using insulating pastes from glasses Nos. 1, 2, 3, 5, and 6 the insulating layer was continuous, shiny, and homogeneous.

The designed openings (concerning particularly small openings) were, however, covered, which certainly disqualified the above mentioned glasses as parent substances for insulating pastes. What is more, the upper conductive paths after heat treatment were displaced with relation to the proposed configuration and often discontinuous. In the case of capacitors which are also located on the test plate, the upper electrode had a series of ply separations and gaps. The reason for the above mentioned flaws was the too low softening point of the parent glasses, not adjusted to the burning temperature 850°C both of the insulating layers and conductive paths. This is evidenced by the fact that these flaws were particularly evident in the case of insulating layers based on glasses Nos. 1, 3, and 5, in which the content of corresponding nucleators of crystallization is half as small as in glasses Nos. 2, 4, and 6. A higher nucleator content causes a greater degree of crystallization and thereby raises the softening point. For this reason also further technological tests were conducted toward increasing the degree of crystallization by way of repeated heat treatment at a temperature of 850°C. Figures 1, 2, and 3 present a picture of upper conductive paths in insulating layers formed sequentially from pastes produced on the basis of glass No. 5 (with a 5% SnO_2 content as the crystallization nucleator), glass No. 6 (with a 10% SnO_2 content) and glass No. 6 after additional heat treatment. The evident, considerable correction both of the appearance of the insulating layer and the upper conductive paths can be connected with the increase in the degree of crystallization connected either with

Picture of upper conductive paths in insulating layers produced sequentially from pastes produced on the basis of:

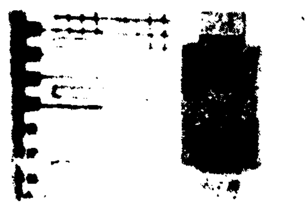


Fig. 1. glass No. 5 (with a 5% SnO_2 content as the crystallization nucleator)

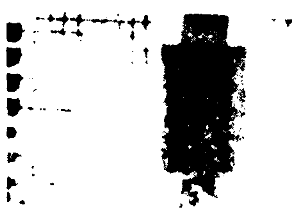


Fig. 2. glass No. 6 (with a 10% SnO_2 content)

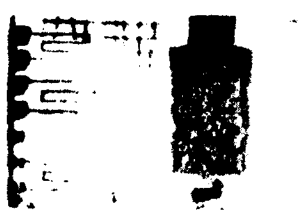


Fig. 3. glass No. 6 (after additional heat treatment)

an increase in the quantity of the nucleating component or with additional heat treatment.

Figures 4 and 5 present X-ray photographs made of glass Nos. 5 and 6. The single crystallic-cassiterite phase nascent in both cases causes an increase in the intensity of lines in the case of glass No. 6 with an increased amount of crystallization nucleators in relation to glass No. 5.

None of the above mentioned glasses, then, satisfies the requirements demanded.

After technological tests for further investigation a paste was rated which was produced on the basis of glass No. 9. That is why the selected properties of this glass will be discussed in detail. Since investigations up to now indicated that the softening point, melting point and deliquescent temperature of glass are very important properties, which, as mentioned earlier, are connected with the degree of crystallization, a thermal differential analysis was made using a derivatograph by the MOM company of Hungary and the characteristic temperatures were determined on a Leitz heat microscope. Measurement conditions on the derivatograph were the following: DTA 1/10, DTG 1/10, TG 200 mg. On the thermogram of glass No. 9 at a temperature of 520°C there appears a very weak exothermic effect of transformation. The first endothermic effect of softening is evident at 560°C, the second corresponding to Littleton's softening point at 700°C. The exothermic effect accompanying crystallization takes place at 800°C. For further investigations samples of powders were used

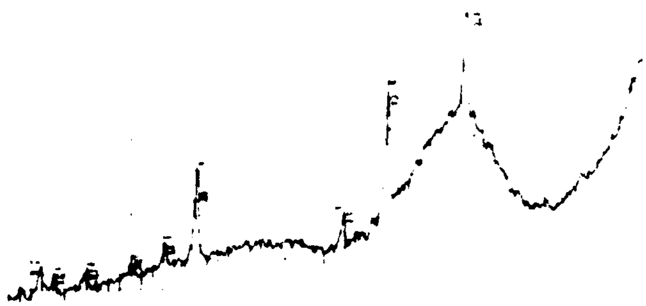


Fig. 4. X-ray photograph of layer produced from glass No. 5

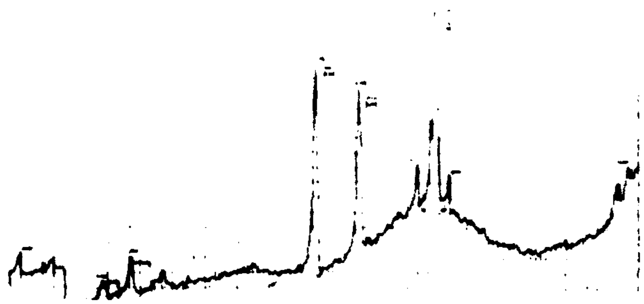


Fig. 5. X-ray photograph of layer produced from glass No. 6

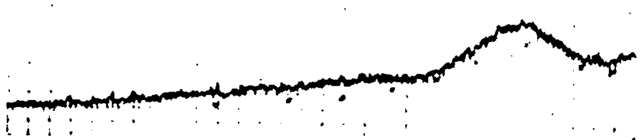


Fig. 6. X-ray photograph of layer produced from glass No. 9 (glass quickly cooled)

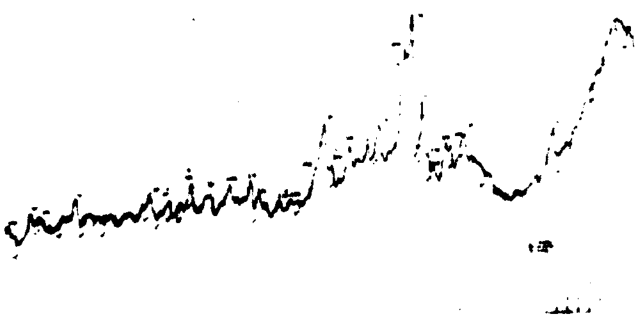


Fig. 7. X-ray photograph of layer produced from glass No. 9 (glass subjected to heat treatment)

which were prepared from glass No. 9, namely:

- from glass quickly cooled by pouring out melted glass mass into water,
- from glass slowly cooled (about 12 hours together with the kiln),
- from glass which, after melting, underwent strictly defined heat treatment, namely retention at $800^{o}C$, that is, at a temperature which, in accord with the results of the differential thermal analysis, corresponds to the crystallization temperature of glass No. 9.

X-ray photograph examinations made on three samples of the above mentioned glass No. 9, prepared in a different way, with a different thermal background, produced on a DRON-1 X-ray machine, indicated that X-ray photographs are identical in the case of the first and second samples. Only the band of the vitreous phase is evident on them (Fig. 6). The X-ray photograph made of the third sampling indicates strong glass recrystallization (Fig. 7). The chief crystallic phases are the lead aluminosilicates:

$$PbO \cdot Al_2O_3 \cdot 6\ SiO_2$$

$$4\ PbO \cdot Al_2O_3 \cdot 2\ SiO_2$$

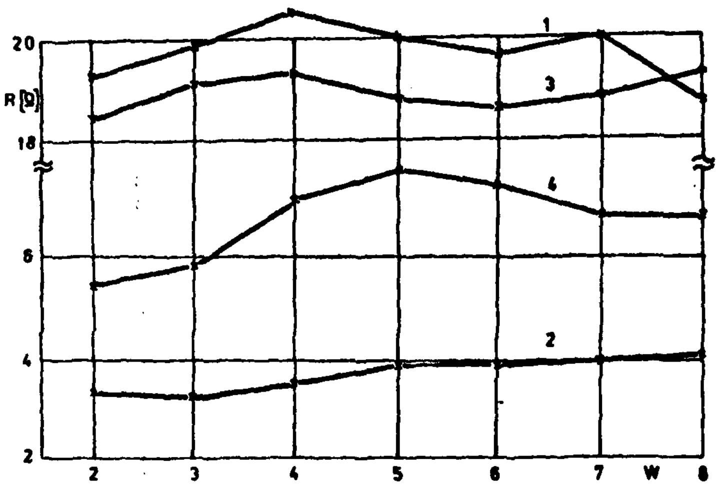
and celsian ($BaO \cdot Al_2O_3 \cdot 2\ SiO_2$) in small quantity. In addition weak lines of willemite (Zn_2SiO_4) were confirmed.

The investigations conducted on characteristic temperatures indicated an increase of about 50°C in the softening, melting and deliquescent temperatures in the case of a sample of recrystallized glass in relation to a sample of amorphous glass.

Taking into consideration the course of the technological process of thick-layered microsystems, whose structure is anticipated by the crossovers of conductive paths as well as several insulating layers, it is calculated that even in the case of a two-layer system the insulating layer can undergo heat treatment even 6 times repeatedly. That is why in determining the optimum content of a crystallic phase in the initial glass consideration is given to the changes in degree of crystallization of the insulating layer sequentially after a 1, 2, 4, and 6 times repeated heat treatment, during which insulating layers are produced from pastes based on amorphous glass or also partly recrystallized glass. Samples were prepared in the following uniform way: the paste was applied by the paint-screen method through a 260 mesh screen onto alum plates measuring 30 x 50 mm. The layer was applied twice in order to avoid incidental defects such as pores or closed air bubbles, after each overprinting the layers were skin-dried, and then baked together in a continuous furnace for a period of 45 minutes, during which the maximum temperature was 850°C. The successive heat treatment processes took place under the same conditions.

A comparison of the X-ray photographs of the insulating layers made on the basis of amorphous glass after 2-, 4-, and 6-times repeated heat treatment made it possible to confirm that in all instances these same crystallic phases appear, during which after each heat treatment the content of the crystallic phase increases. A comparison of the X-ray photographs for analogical insulating layers made out of glass partly recrystallized indicates a similar phenomenon, however the increase in the degree of crystallization is slower. Experimentally it was confirmed that the best results are obtained using glass partly recrystallized (heat treatment of glass at 800°C for 2 hours); after a 6th time heat treatment of the insulating layer there is then achieved about a 40% degree of crystallization. This same content of the crystallic phase is reached after heat treatment of glass for 5 hours at 800°C. With an increase in degree of crystallization of the insulating layer its surface roughness changes; after each successive heat treatment process (examinations were conducted to 6 firings) a considerable decrease in the roughness of the insulating layers can be observed, which can be attributed to the increase in degree of crystallization.

Further investigations were aimed at determining the dependence of selected electric parameters of the insulating layers on the amount of successive heat treatment processes on the layers, and what happens after, on the degree of crystallization.



conductive path 0. 3 mm. wide

1 - *at the base*

2 - in insulating layer

conductive path 0. 5 mm. wide

3 - *at the base*

4 - in insulating layer

Fig. 8. Resistance changes of conductive paths in function of successive firings

The following investigations were conducted:

- determining resistance change of the conductive paths located in the insulating layer in comparison with changes of conductive paths placed directly on an alum base, in the function of successive heat treatment processes--Fig. 8,

- determining the change in the tangent of the angle of loss of the insulating layers in the function of successive heat treatment processes--Fig. 9,

Fig. 9. Tg of angle of loss of insulating layers in function of successive heat treatment processes

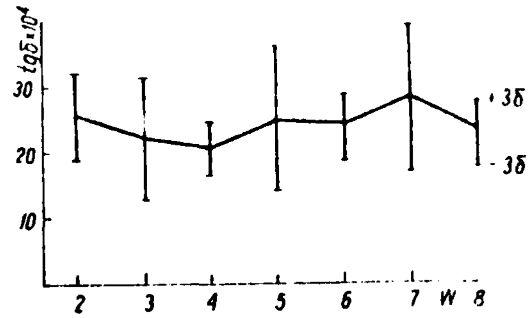
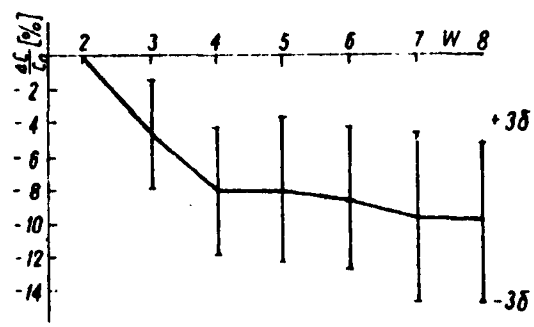


Fig. 10. Capacitance of insulating layers in function of successive heat treatment processes



- determining the change in the capacity of the insulating layers in the function of the successive heat treatment processes--Fig. 10.

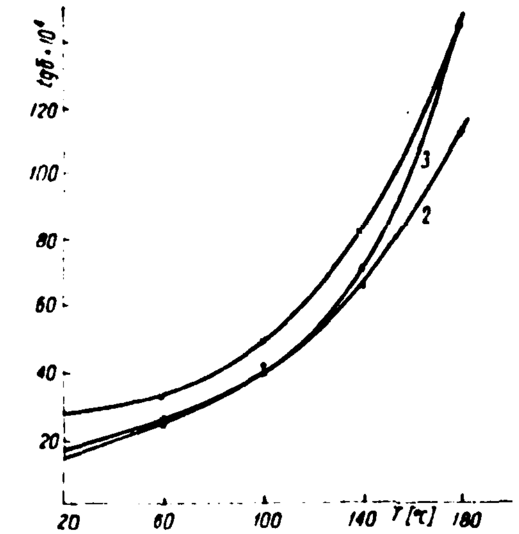
The graphs presented indicate that both the resistance of the conductive paths and the tangent of the angle of loss of the insulating layers to a small degree only depend on the number of firings. The capacity of the insulating layers decreases, on the other hand, within the limits of the increase in the number of firings, which can be attributed to the increase of the degree of crystallization.

Testing was also done aimed at determining the influence of size and shape of the granules of glass powder on the basic properties of the insulating layers. It was shown that good results can be reached using the addition to the glass powder having granules of irregular shape of the selected fraction of granules having a round shape. The result of the above mentioned supplementary testing was to get more accurate parameters of the technological process of preparing the parent substances and technological process of preparing the insulating paste for the crossovers and multilayered circuits. In order to check the accuracy of the elaborated technological process of preparing the insulating paste and the insulating layers obtained an information run was made of the test plates and testing was conducted. Their results, contained in Table 1, allow for a comparison of selected properties of the test layers made on the basis of various insulating pastes with catalog data from foreign companies. Independent of the parameters collected in the table, there are defined:

- the temperature dependence of the tangent of the angle of loss-Fig. 11
- the temperature dependence of capacitance-Fig. 12
- dielectric constant - frequency function-Fig. 13
- tg of the angle of loss in frequency function-Fig. 14
- capacitance in time function-Fig. 15
- tg of the angle of loss in time function-Fig. 16

Selected properties of layers from insulating layers

Type of insulating paste used	Dielectric constant f = 1 kHz	Tg of angle of loss [%]	Insulation resistance [Ω]	Break-down strength [V]
Paste based on powder of glass No. 9 (with irreg. gran.)	7	0,25	10^{12}	500
Paste based on powder of glass No. 9 (with 20% addit. of glass powder with round granules)	6	0,13	10^{13}	500
DuPont 8190 paste (catalog data)	6-9	0,5	10^{10}	500
DuPont 9429 paste (catalog data)	9-12	0,05-0,25	10^{12}	400



1 - No. 9 glass powder with irregular granules

2 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules - 1 layer

3 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules - 2 layers

Fig. 11. Temperature of dependence of tg of angle of loss of insulating layers

The supplementary investigations conducted give a fuller picture of the behavior of the insulating layers both during the technological process and under service conditions.

1 - No. 9 glass powder with irregular granules

2 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules - 1 layer

3 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules - 2 layers

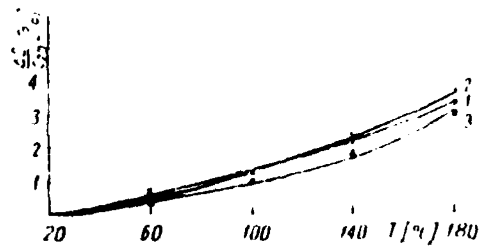
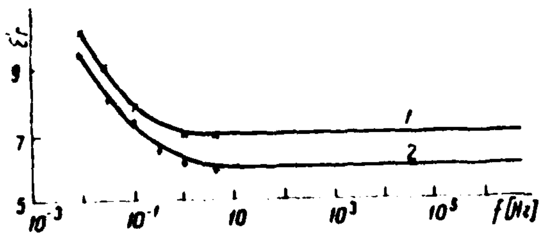


Fig. 12. Capacitance of insulating layers depending on temperature



1 - No. 9 glass powder with irregular granules

2 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules

Fig. 13. Dielectric constant of layers in frequency function

1 - No. 9 glass powder with irregular granules

2 - No. 9 glass powder with irregular granules with 20% addition of No. 9 glass powder with round granules

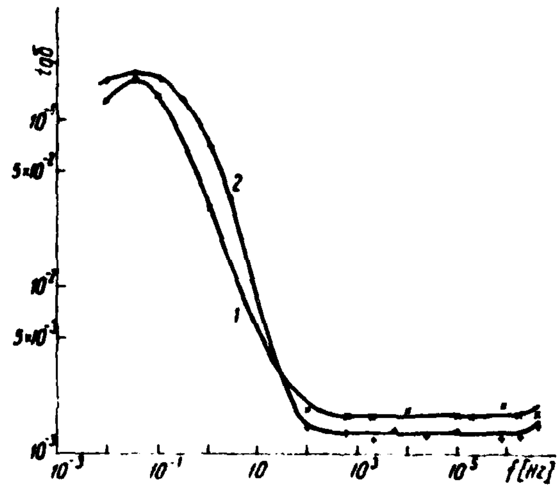


Fig. 14. Tg of angle of loss of layers in frequency function

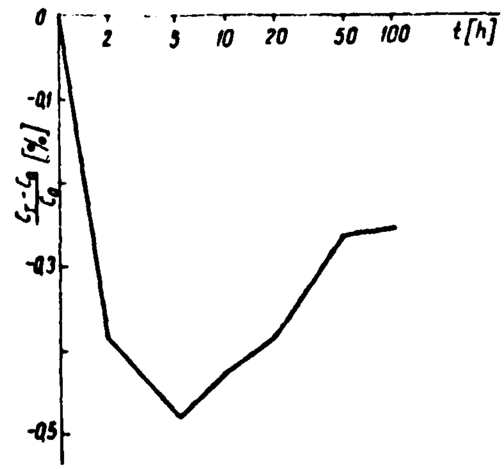


Fig. 15. Capacitance of insulating layer in time function

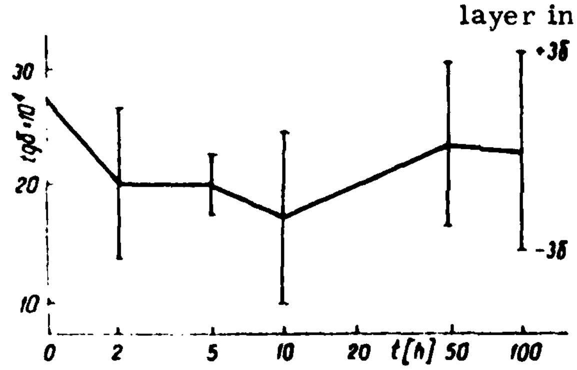


Fig. 16. Tg of angle of loss in time function

Recapitulating, the results achieved from the testing concerning the basic parameters of the insulating layers make it possible to confirm that the elaborated paste can be used for thick-layered systems including the crossovers of conductive paths.

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